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Inter-laboratory Comparisons Small-Scale Safety and Thermal Testing of Improvised Explosives

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Variation of Methods in Small-Scale Safety and Thermal Testing of Improvised Explosives

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Abstract

One of the first steps in establishing safe handling procedures for explosives is small-scale safety and thermal (SSST) testing. To better understand the response of homemade or improvised explosives (HMEs) to SSST testing, 16 HME materials were compared to 3 standard military explosives in a proficiency-type round robin study among five laboratories, two U. S. Department of Defense and three U. S. Department of Energy, sponsored by the Department of Homeland Security, Science & Technology Directorate, Explosives Division. The testing included impact, friction, electrostatic discharge (ESD) and thermal. The testing matrix has been designed to address problems encountered with improvised materials: powder mixtures, liquid suspensions, partially wetted solids, immiscible liquids, and reactive materials. All testing materials and/or precursors came from the same batch distributed to each of the participants and were handled, pretreated and mixed by standardized procedures.

For this proficiency test, the participants had similar equipment, usually differing by vintage. This allowed for a direct comparison of the results from each participant to the average of the results from all the participants. Some general trends observed for each series of tests were: 1) Drop hammer—LLNL usually found the materials less sensitive than the average with materials that have high sensitivity to impact and LANL usually found the materials less sensitive than the average with materials that have high sensitivity to impact; 2) friction—LLNL found the materials less sensitive than the average; 3) and ESD—IHD usually found the materials less sensitive than the average.

In this report, the proficiency test data from all the participants is compared and contrasted for impact, selected friction and ESD testing. Other friction and thermal data will be addressed elsewhere as well as the statistical analysis of several repeated measurements on the proficiency test standards.

Keywords: Small-Scale Safety Testing, Homemade Explosives, Improvised Explosives, Impact, Friction, Electrostatic discharge

1 Introduction

Small-Scale Safety and Thermal (SSST) testing is one of the first steps in developing safe handling practices for energetic materials [1,2]. These tests are designed to determine sensitivity of energetic materials to handling and storage conditions—drop hammer for impact sensitivity; friction for shear force sensitivity; electrostatic discharge (ESD) for spark or static sensitivity; differential scanning calorimetry (DSC) for thermal stability; many others tests for specific types of reactivity.

SSST testing methods were developed for primarily safe handling of military and mining explosives. With the increased interest in understanding improvised or homemade explosives (HMEs), SSST testing methods have been applied to various mixtures and pure materials that make up this class of energetic materials.

Performance evaluation of HMEs has been on the rise in recent years [3]. With this activity comes the need to apply SSST methods to develop safe handling protocols for HMEs. Often, HMEs are formed by mixing oxidizer and fuel precursor materials, and typically, the mixture precursors are combined shortly before use. The challenges to produce a standardized inter-laboratory sample are primarily associated with mixing and sampling. For solid-solid mixtures, the challenges primarily revolve around adequately mixing two powders on a small scale, producing a mixture of uni-form composition—particle size and dryness often being a factor. For liquid-liquid mixtures, the challenges revolve around miscibility of the oxidizer with the fuel causing the possibility of multiphase liquid systems. For liquid-solid mixtures, the challenges revolve around the ability of the solid phase to mix completely with the liquid phase, as well as minimizing the formation of intractable or ill-defined slurry-type products. For all these mixtures, taking a representative sample is a barrier to adequate testing.

The Integrated Data Collection Analysis Program (IDCA), a Department of Homeland Security, Explosives Division funded effort, has been conducting SSST testing on a series of HME or improvised explosives, utilizing standard SSST testing practices as applied to military explosives [4]. The testing has been by a round-robin or proficiency test where 19 HMEs and military explosives have been tested by three U.S. Department of Energy and two U.S. Department of Defense Laboratories—Lawrence Livermore National Laboratory (LLNL), Los Alamos National Laboratory (LANL), Sandia National Laboratories (SNL), Naval Surface Warfare Center, Indian Head (IHD), and Air Force Research Laboratory, Tyndall Air Force Base (AFRL). This paper compares the testing results from these laboratories for impact (Type 12 or modified Type 12), friction (BAM) and electrostatic discharge (ABL and custom built). Comparison of BAM and ABL friction results, thermal results and issues, and statistical analyses of the proficiency test will be reported elsewhere. Preliminary results have been reported previously [5].

2 Results

The objectives of the IDCA effort are to accumulate SSST testing data on several HMEs, compare data from several laboratories taken on the same materials, and potentially derive statistical significance of the findings for both inter- and intra-laboratory performance. In order to achieve this, as many variables as possible in the testing were eliminated. Each component material was distributed to all the performers and the mixtures (when appropriate), were prepared, pretreated, and stored according to strict standardized procedures [6]. Test methods at the individual laboratories were modified to some extent, but each laboratory was allowed to use their own standard operating procedures when testing. In addition, no additional environmental controls were applied, so parameters, such as temperature and humidity, of testing varied throughout the testing and location. Data reduction methods selected were a modified Bruceton up-down method and Neyer D-Optimal method for impact testing [7,8], modified Bruceton and Threshold Initiation Level (TIL) for friction [7,9], and TIL for ESD [9]. Each laboratory had a choice how to perform these analyses, so there were minor variations. For the modified Bruceton method, the variations were the choice of linear or log spacing. For TIL, the variation was the number of testing attempts. Detection methods for positive or negative reactions varied among the participants, although all participants used at least operator input in addition to other methods. For impact testing, all used sound and visible detection by the operator. In addition, LLNL and LANL employed microphones linked to a sound meter. For friction, all used sound and visual detection by the operator. For ESD, all used sound and visual detection by the operator while LLNL and SNL also used gas detection meters.

Due to the massive amount of data accumulated, only summary figures and tables are presented here. A full listing of the data can be found in the Supplementary files available separately from the corresponding author [10].

2.1 Materials

Table 1 lists the materials used in the proficiency test, along with the abbreviations used throughout the text and the form of the material upon mixing.

Table 1. IDCA mixtures and pure materials formulations

Material[a]	ID	Form[b]
$KClO_4$/Al	$KClO_4$/Al	Dry powder
$KClO_4$/C[c]	$KClO_4$/C	Dry powder
$KClO_4$/dodecane	$KClO_4$/D	Wet powder
$KClO_3$/dodecane	$KClO_3$/D	Wet powder
$KClO_3$/sugar[d]	$KClO_3$/Sg 100	Dry powder
$KClO_3$/sugar[e]	$KClO_3$/Sg AR	Dry powder
$NaClO_3$/sugar[d]	$NaClO_3$/Sg	Dry powder
AN[f]	AN	White powder
Bullseye® gunpowder	GP	Black powder
AN/Bullseye® gunpowder	AN/GP	Gray powder
UNi/Al[g]	UNi/Al	Dry powder
UNi/Al/S	UNi/Al/S_8	Dry powder
H_2O_2/cumin[h,i]	H_2O_2/Cmn	Viscous paste
H_2O_2/nitromethane[j]	H_2O_2/NM	Miscible liquid
H_2O_2/flour[h,k]	H_2O_2/Fl	Sticky paste
H_2O_2/glycerol[h]	H_2O_2/Gl	Miscible liquid
HMX Grade B	HMX	Powder
RDX Type II Class 5[l]	RDX	Powder
PETN Class 4[l]	PETN	Powder

[a] Mixture or pure material, [b] observed physical form, [c] activated charcoal (Darco), [d] icing sugar + -100 mesh $KClO_3$, [e] icing sugar + as received $KClO_3$, [f] ammonium nitrate, [g] Urea nitrate, [h] 70% H_2O_2, [i] *cuminum cyminum*, [j] 90% H_2O_2, [k] chappati, [l] standard

The materials listed in the table span a wide range of compositions and physical forms, some not encountered in normal SSST testing. Examples are: 1) H_2O_2/fuel mixtures, due to the reactivity of the peroxide, can form intractable pastes that continually change (and are dangerous); 2) solid-solid mixtures can be compose of mismatched particle sizes where localized oxidizer to fuel ratios are significantly different that the bulk; 3) liquid-solid mixtures have volatile components that evaporate during the analysis changing the oxidizer to fuel mole ratio. All these are problematic and complicate obtaining representative and reproducible samples.

2.2 Impact Testing Results

Impact testing is used to evaluate the sensitivity of the energetic material to non-shock initiated reactions caused by dropping or having something dropped on it. The test is simple—the sample is placed in a holder (anvil) and a force is applied by dropping a weight at increasing heights until a reaction occurs.

All materials in Table 1 were examined for impact sensitivity by the drop hammer method. Three of the participants tested the full suite of materials, and two of the participants tested a subset. The full results are listed in supplementary material. Each material was examined usually in triplicate or more. In some cases, different sandpapers were used by LLNL and LANL. The values in the figures below are averages of all the values with a specific sandpaper type for each listed material. Pelletized results were excluded from the averages and are discussed below.

2.2.1 Comparison of Results from Participants

Figures 1 and 2 show graphs of the impact data from the individual participants compared to the average of all the participants for each material. The values are the DH_{50}, in cm, by a modified Bruceton method, load for 50% probability of reaction. The red line is the average data and the symbols are Individual laboratory data. (Note: values above the line indicate less sensitivity than the average and value below the line indicate higher sensitivity than the average.) The apparatus used by each participant: LANL, LLNL, IHD, Type 12; AFRL, SNL, MBOM with Type 12 tooling. There are two graphs, one for DH_{50} values that indicate a material reasonably sensitive to impact, from approximately 10 to 50 cm, and the other is for DH_{50} values that indicate a material with low sensitivity to impact, from 90 to 170 cm. The highest DH_{50} value reflects the drop weight height limits for the participants, in cm: LLNL, 170; LANL, 320; IHD, 320; AFRL, 116; SNL, 115. There were other materials that exhibited DH_{50} values above 170 cm. However, for comparison purposes, the 170 cm height was deemed as the cutoff value.

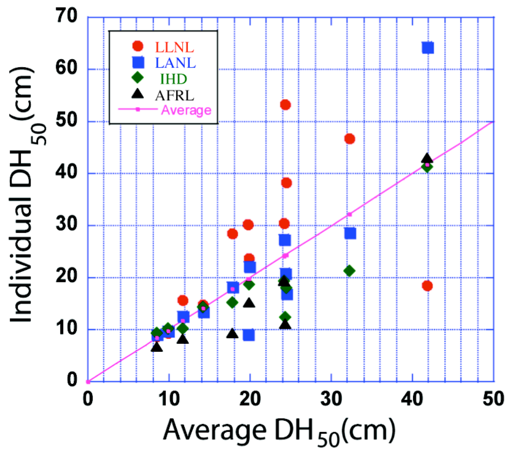

Figure 1. Impact data (DH_{50}, in cm) for the average of all participants vs. the average of each of the participants in range of 0 to 50 cm

The results in Figure 1 for the 10 to 50 cm range show: LLNL (red circles) values mostly are above the average line for DH_{50} values below 40 cm; LANL (blue squares) values generally are below the average line for DH_{50} below 40 cm; IHD generally tracks LANL values, but show slightly higher corresponding sensitivity; AFRL values generally reports the highest sensitivity of the all the participants for a specific material. The results in Figure 2 for the 90 to 170 cm range show: LLNL and AFRL values are below the average; LANL values are above the average: IHD values do not exhibit a trend.

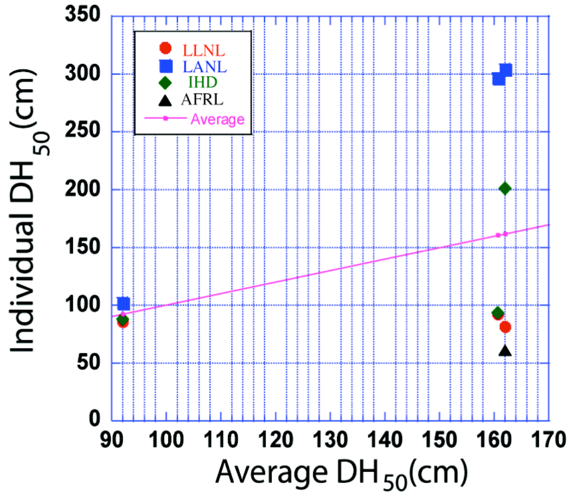

Figure 2. Impact sensitivity data (DH_{50} in cm) for the average of all participants vs. the average of each of the participants in range of 90 to 170 cm

Table 2 shows the relative ranking of the *solid* materials tested for impact sensitivity by LLNL, LANL, IHD, and AFRL based on DH_{50} values. AFRL did not participate in all of the testing, so some materials are missing from the relative ranking. Although there are specific differences in the relative rankings comparing individual participant results, in general, the rankings can be divided into 3 sensitivity groups—PETN, middle sensitive, and insensitive. Note that the insensitivity is defined differently for each participant because of equipment configuration (drop height maximum).

Table 2. Relative Ranking of Impact Sensitivity for Selected Solid Mixtures[1,2]

LLNL	LANL	IHD	AFRL[3]
PETN	PETN	PETN	PETN
$KClO_3$/D	$KClO_3$/D	$KClO_3$/D	$KClO_3$/Sg AR
$KClO_3$/Sg 100	$KClO_3$/Sg AR	$KClO_3$/Sg AR	$NaClO_3$/Sg
$KClO_3$/Sg AR	$KClO_3$/Sg 100	GP	GP
KP/Al	HMX	$KClO_3$/Sg 100	RDX
RDX	GP	$NaClO_3$/Sg	
$NaClO_4$/Sg	RDX	HMX	
$KClO_4$/D	$NaClO_3$/Sg	RDX	
HMX	KP/D	$KClO_4$/D	$KClO_4$/D
AN/GP	AN/GP	AN	$KClO_4$/Al
GP	KP/Al	AN/GP	AN
AN		$KClO_4$/Al	
$KClO_4$/C *UNi/Al* *UNi/Al/S_8*	*AN* *$KClO_4$/C* *UNi/Al* *UNi/Al/S_8*	*$KClO_4$/C* *UNi/Al* *UNi/Al/S_8*	*$KClO_4$/C*

1. For abbreviations see Table 1; 2. Materials in italics showed no sensitivity at the upper testing limits of the equipment; 3. AFRL performed limited testing.

All participants found PETN to be the most sensitive material. The $KClO_3$ mixtures are all rated relatively highly sensitive. $KClO_4$/D, RDX, $NaClO_3$/Sg and HMX fall into the middle and AN/GP fall below those. GP and $KClO_4$/Al vary widely depending upon the participant. $KClO_4$/C, UNi/Al, and UNi/Al/S_8 exhibit no sensitivity. AN varies on the low end of sensitivity, and exhibits none for LANL.

The *liquid* mixtures were also ranked according to relative sensitivity. They were not included in Table 2 because the testing protocols were different than for the powders. LANL used a bare anvil with a custom-built design [11,12] that employs a ring of grease to contain the liquid sample and a magnet that holds the striker 3 mm above the liquid sample before testing. LLNL also used the grease on the same anvil as used for solid samples and uses a 1-kg striker placed on top of the grease ring before testing. In addition, LLNL and IHD used sandpaper in selected cases. IHD also used the Cavity Drop cell, ASTM D 2540-93 [11,13,14], that has a closed cavity for holding the liquid sample in place.

Table 3 shows the DH_{50} results from the H_2O_2-based mixtures. Except for H_2O_2/NM mixture, LLNL found the mixtures insensitive when using the bare anvil. Even though LANL found all the mixtures sensitive using the bare anvil, only H_2O_2/NM exhibited any significant sensitivity (slightly less than PETN at 8.1 ± 0.6 cm). Both IHD and LLNL found the materials to be sensitive when using sandpaper to hold the mixtures in place. Using the Cavity Drop cell, IHD found the materials not to have sensitivity. However, the limiting drop height for this cell is 50 cm.

Table 3. DH_{50}, in cm, Impact Sensitivity of H_2O_2 Mixtures

Material[1]	LLNL BA[2]	LANL BA[3]	LLNL 180[4]	IHD 180[4]	IHD CD[5]
H_2O_2[8]/Cmn	> 177[6]	109.2 ± 6.6	86.0 ± 1.0	18.0 ± 1.0	> 50[7]
H_2O_2[8]/Fl	> 177[6]	292.8 ± 4.9	92.6 ± 2.1	93.0 ± 28.7	NA
H_2O_2[8]/Gl	> 177[6]	132.3 ± 6.1	NA[10]	NA[10]	> 50[7]
H_2O_2[9]/NM	30.3 ± 3.1	9.3 ± 1.0	NA[10]	NA[10]	> 50[7]

1. See Table 1 for abbreviations; 2. BA = bare anvil, 1-kg striker weight; 3. Bare anvil, 0.8-kg striker weight; 4. 180-grit garnet dry sandpaper on anvil; 5. Cavity Drop cell; 6. 177 cm is the drop height limit of LLNL drop hammer; 7. 50 cm is the drop height limit of IHD Cavity Drop cell; 8. 70% H_2O_2; 9. 90% H_2O_2. 10. Did not test.

2.2.2 Variability Caused by Sandpaper

Table 4. Variability in Impact Sensitivity of Selected Materials Relative to RDX[1] Based on Sandpaper

Material	Lab	120	150	180
$KClO_3$/D	LLNL	14.4		-12.5
$KClO_4$/Al	LLNL	152.9[2]		-4.9
$KClO_4$/D	LLNL	152.9[2]		8.7
PETN	LLNL	-13.3		-13.5
AN	LLNL	131.9		60.2
AN/GP	LLNL	61.7		25.0
HMX	LLNL	21.9		16.4
$KClO_3$/Sg 100	LANL		-8.4	-10.2
$KClO_3$/Sg AR	LANL		-10.4	-10.4
$KClO_3$/D	LANL		-14.2	-12.7

1. Values determined by Bruceton DH_{50}, in cm, relative to RDX measured with the same sandpaper; 2. DH_{50} value higher than the highest drop weight level (177 cm) for LLNL.

In the drop hammer test, Type 12A testing requires the use of sandpaper. The sample is placed on the sandpaper, then positioned on the anvil where the striker weight is usually placed on top of the sample, at least for solids. The testing is complete when the drop weight impacts on the striker weight. Table 4 shows the sensitivity of selected materials in this study from testing using the different sandpaper to hold the sample. DH_{50} values are shown relative to the RDX standard measured with the corresponding sandpaper. The sandpaper is delineated by grit size. LLNL performed testing on selected materials using both 120-grit Si/C sandpaper and 180-grit garnet sandpaper (IDCA standard). LANL performed testing on selected materials using 150-grit and 180-grit (IDCA standard) garnet sandpapers.

For mixture $KClO_3$/D, $KClO_4$/Al, $KClO_4$/D, AN, AN/GP, LLNL data shows a very large discrepancy between the results using 120-grit Si/C and corresponding 180-grit garnet sandpapers. The military materials, PETN and HMX, show some, but little differences. For the $KClO_3$/Sg mixtures and $KClO_3$/D, LANL data show slight differences between the results from the 150- and the corresponding 180-grit sandpapers.

2.2.3 Neyer D-Optimal and Bruceton Comparison

LANL performed impact testing by both Bruceton and Neyer D-Optimal analysis protocols of all the materials. Figure 3 compares these results for materials that exhibited sensitivity up to the 40 cm drop height.

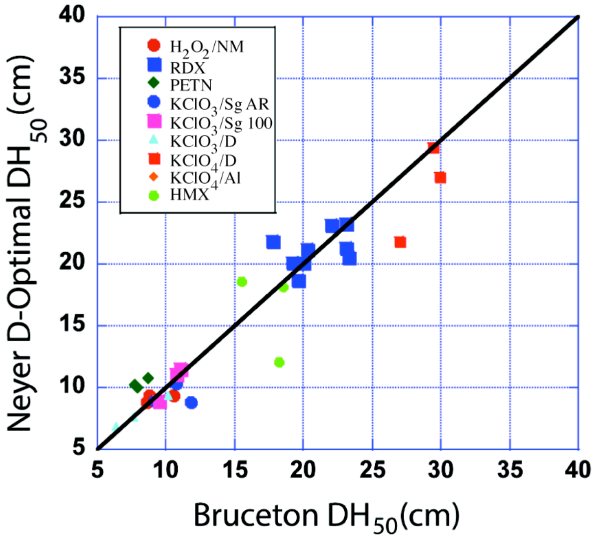

Figure 3. Comparison of DH_{50} values determined by the modified Bruceton method to Neyer D-Optimal method

The agreement of the two methods is clear from the figure. Most determinations for a specific material fall around the 1 to 1 correlation line. Only PETN shows some discrepancy from the 1 to 1 correlation. This discrepancy is discussed below.

2.2.4 Powder and Pressed Samples

Table 5. Impact Sensitivity Comparing RDX and HMX in Pellet and Powder Form

Form[1,2]	T, °C	RH, %[3]	DH_{50}, cm[4]	s, cm[5]	s, log unit[5]
RDX Pellet (120)[6]	24	18	28.8	2.8	0.042
RDX Powder (120)[6]	23	22	24.2	0.8	0.015
RDX Powder (120)[6]	23	23	22.0	1.9	0.035
RDX Pellet (120)[7]	24	32	34	4.63	0.059
RDX Powder (120)[8]	24	18	24.8	3.09	0.054
HMX Pellet (120)	24	18	46	9.38	0.088
HMX Pellet (180)	23	20	41	1.13	0.012
HMX Powder (180)	25	18	38	3.07	0.035

1. Pellet is pressed in a commercial powder press, powder is loose flowing powder; 2. Value in parentheses: 120 is 120-grit Si/C wet sandpaper, 180 is 180-grit garnet dry sandpaper; 3. Relative humidity; 4. Values determined by Bruceton DH_{50}, in cm; 5. Standard deviation; 6. From RDX-1 (see supplementary material); 7. From RDX-2 (see supplementary material); 8. From RDX-3 (see supplementary material).

LLNL determined the impact sensitivity of selected materials in two different forms—pelletized and free-flowing powder. The pelletized form was selected because many military-type explosives are routinely tested in this form. Table 5 shows the DH_{50} results for the RDX standard and HMX. For both materials, the pressed pellets exhibit less sensitivity than the corresponding loose powders.

2.3 Friction Testing Results

Friction sensitivity was measured in the proficiency test by testing on BAM and ABL equipment. Reported here are the friction testing results on the BAM equipment. The results for the ABL equipment will be reported elsewhere with a comparison analysis of these materials on both the ABL and BAM equipment [15]. Friction testing is used to evaluate non-shock initiated reactions due to the energetic material being subjected to abrasion. For the BAM equipment test, the material is staged on a movable porcelain or ceramic porous surface that is actuated under a stationary stylus. The force on the pin is increased until a reaction occurs. The full results are listed in supplementary material.

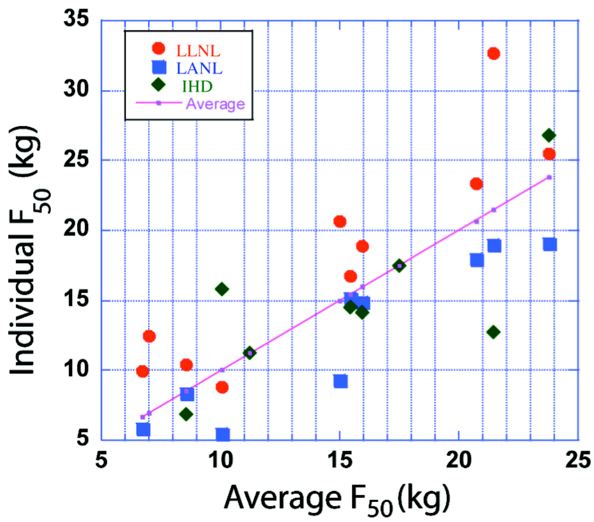

Figure 4. Friction sensitivity by modified Bruceton method (F_{50}, in kg) for the average of all participants vs. the average of each of the participants taken on BAM testing equipment

Figure 4 shows the average friction data from the individual participants for each material compared to the average of all the participants for each material. The BAM measurements are on equipment (different vintages) at LLNL, LANL, and IHD laboratories. AFRL does not have a BAM apparatus. F_{50}, in kg, is by a modified Bruceton method, load for 50% probability of reaction. The red line is the average of all the data, in kg, and the symbols are the individual lab data.

The results in Figure 4 show: LLNL (red dots) always derives a value for F_{50} above the corresponding average value for each of the materials (except in one case); LANL (blue squares) always derives a value for F_{50} below the corresponding average value for each of the materials; IHD (green diamonds) values tend to be around the corresponding average values. LLNL finds the materials to be less sensitive.

Table 6 lists the relative order of the materials for friction sensitivity determined by the modified Bruceton method on the BAM equipment. LLNL and LANL identify about the same relative sensitivity, although in a slightly different order. In addition, the same materials are identified as insensitive. The first four most sensitive materials are the same, and the next four materials are the same, although in different order, and so on.

Table 6. BAM Friction Sensitivity Relative Ranking by Bruceton Friction Method, F_{50}, for LLNL, LANL, and IHD[1,2]

LLNL	LANL	IHD
$NaClO_3$/Sg	$KClO_3$/Sg AR	$KClO_3$/Sg AR
$KClO_3$/Sg 100	$NaClO_3$/Sg	$KClO_3$/Sg 100
PETN	$KClO_3$/Sg 100	PETN
$KClO_3$/Sg AR	PETN	H_2O_2/Cmn
$KClO_4$/Al	GP	AN/GP
HMX	HMX	HMX
GP	$KClO_4$/Al	$KClO_4$/Al
RDX	RDX	$NaClO_3$/Sg
$KClO_3$/D	AN/GP	H_2O_2/Gl
AN/GP	$KClO_3$/D	RDX
		$KClO_3$/D
$KClO_4$/D	*$KClO_4$/D*	
$KClO_4$/C	*$KClO_4$/C*	*$KClO_4$/C*
AN	*AN*	*AN*
H_2O_2/Cmn	*H_2O_2/Cmn*	
H_2O_2/Fl	*H_2O_2/Fl*	*H_2O_2/Fl*[3]
H_2O_2/Gl	*H_2O_2/Gl*	
H_2O_2/NM	*H_2O_2/NM*	*H_2O_2/NM*[3]
UNi/Al	*UNi/Al*	*UNi/Al*
UNi/Al/S_8	*UNi/Al/S_8*	*UNi/Al/S_8*

1. For abbreviations see Table 1; 2. Materials in italics showed no sensitivity at the upper testing limits of the equipment; 3. IHD did not test by Modified Bruceton

The relative sensitivity order determined by IHD is somewhat different. However, if the two H_2O_2 mixtures are removed from the list, the relative sensitivity order is about the same as determined by LLNL and LANL, except for the placement of $NaClO_3$/Sg.

Table 7 lists the friction values determined on the BAM equipment using the TIL friction method by LLNL, LANL, and IHD. SNL measured the TIL values for two materials, RDX and PETN and found, in kg, 16.3 ± 0.5 and 3.3 ± 0.2, respectively. The following summarizes the TIL results, for the:

- Military explosives (RDX, PETN, and HMX), LLNL always finds the material less sensitive that the others; LANL usually finds the material less sensitive than IHD, and IHD usually finds the material more sensitive than the others.
- Mixtures that are solids ($KClO_3$/Sg 100, ($KClO_3$/Sg AR, $KClO_3$/D, $KClO_3$/Al, $KClO_4$/D, $KClO_4$/C, $NaClO_3$/Sg, AN, GP, AN/GP, UNi/Al, UNi/Al/S_8), LLNL shows the material to be less sensitive than both LANL and IHD for most materials; LANL and IHD vary position on sensitivity; only IHD found the UNi mixtures sensitive.
- Mixtures than are liquids or viscous (H_2O_2/Cmn, H_2O_2/Fl, H_2O_2/Gl, H_2O_2/NM), only IHD found any of the materials sensitive; LLNL and LANL did not register any sensitivity within the limits of their equipment.
- Mixtures that were found insensitive to the limits of the equipment ($KClO_4$/D, $KClO_4$/C, AN, H_2O_2/Cmn, H_2O_2/Fl, H_2O_2/Gl, H_2O_2/NM, UNi/Al, UNi/Al/S_8), LLNL and LANL

always agreed; IHD often did not agree and usually found the materials moderately sensitive (not just marginally sensitive).

- Mixtures with sugar as the fuel ($KClO_3$/Sg 100, $KClO_3$/Sg AR, $NaClO_3$/Sg), the sensitivity order determined is LLNL < IHD < LANL.

Table 7. BAM Friction Sensitivity as Measured by TIL Method for LLNL, LANL, IHD.[1,2,3]

Material	LLNL, in kg	LANL, in kg	IHD, in kg
RDX 1	19.2 ± 2.4	19.2	15.1 ± 0.7
RDX 2	18.0 ± 1.2	10.4 ± 1.4	11.8 ± 0.7
RDX 3	16.8	11.4 ± 1.4	11.4 ± 0.7
RDX 4	16.3 ± 0.5	13.9 ± 1.4	11.8 ± 0.6
PETN	6.4 ± 0.8	4.9	4.7 ± 0.9
HMX	13.1 ± 1.2	12.2	8.6
$KClO_3$/Sg 100	6.8 ± 1.1	2.4	2.0 ± 0.6
$KClO_3$/Sg AR	8.8 ± 2.1	< 2.4[4]	2.8 ± 0.2
$KClO_3$/D	12.3 ± 0.9	7.2	2.8 ± 0.2
$KClO_4$/Al	9.6 ± 1.2	7.2	12.2 ± 2.5
$KClO_4$/D	> 36[5]	> 36[5]	33.1 ± 3.7
$KClO_4$/C	> 36[5]	> 36[5]	> 36.7[5]
$NaClO_3$/Sg	6.1 ± 0.2	2.4	4.4 ± 1.6
AN	> 36[5]	> 36[5]	> 36.7[5]
GP	16.4 ± 1.8	5.6 ± 1.4	13.9 ± 1.4
AN/GP	27 ± 2.5	13.0 ± 1.4	12.2
H_2O_2/Cmn	> 36[5]	> 36.7[5]	8.6
H_2O_2/Fl	> 36[5]	> 36.7[5]	11.4 ± 0.7
H_2O_2/Gl	> 36[5]	> 36.7[5]	11.8 ± 0.7
H_2O_2/NM	> 36[5]	> 36.7[5]	> 36.7[5]
UNi/Al	> 36[5]	> 36.7[5]	> 36.7[5]
UNi/Al/S_8	> 36[5]	> 36.7[5]	13.1 ± 1.2

1. Threshold Initiation Level (TIL) is the load (kg) at which zero reactions out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level; 2. Average of individual values ± standard deviation; 3. Numbers without ± values indicate all the same value was measured; 4. Sensitivity too high for instrument to measure; 5. Sensitivity too low for instrument to measure.

2.4 Electrostatic Discharge Testing Results

ESD testing is used to evaluate the non-shock initiated reactions due to the energetic material becoming exposed to spark or static discharge. The test is simple, the material is located between two electrodes and increasingly energetic sparks are discharged until a reaction is detected. In the proficiency test, two ESD systems were used, the commercially available ABL system (differing vintages) and a custom built system by LLNL. Except where noted, all the data compared below were derived from comparable ABL systems. The full results are listed in the supplementary material.

2.4.1 Comparison of Performance

Figure 5 shows the ESD TIL data taken by the ABL equipment. TIL is the load (joules) at which zero reactions out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher energy level. The averages of individual data of each participant are represented vs. average data of all the participants (1:1 is red line). LLNL (red dots) and AFRL (black triangles) have limited data sets with the ABL device. In general, IHD (green diamonds) report lower sensitivities compared to LANL (blue squares).

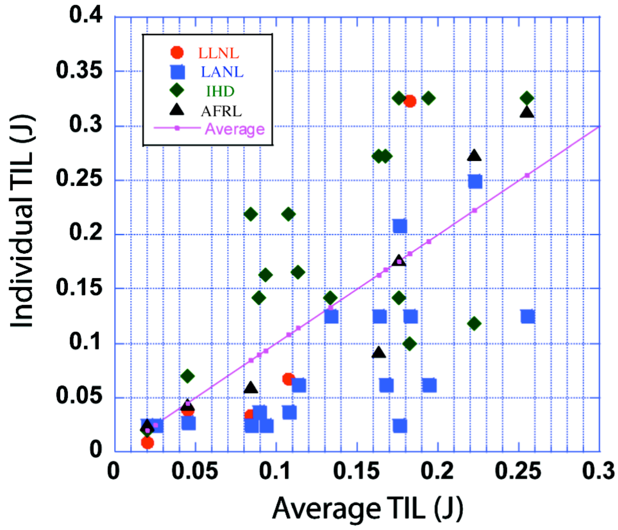

Figure 5. ESD sensitivity data (TIL in J) for the average of all participants vs. the average of each of the participants taken on ABL testing equipment

Table 8 shows the average TIL values for the ESD measurements. All the data shown were from measurements with no additional resistance incorporated in the circuit. LLNL, using a custom made ESD system with a 510-Ω resistor in the circuit (to mimic the human body), measured sensitivity of multiple materials (RDX 1, RDX 2, $KClO_3$/Sg 100, $KClO_3$/Sg AR, $KClO_3$/D, $NaClO_3$/Sg, AN, GP, H_2O_2/Cmn, H_2O_2/Fl, H_2O_2/Gl H_2O_2/NM). This is discussed below. During the proficiency test, LLNL also brought on-line an ABL ESD system and was able to complete testing at 0-Ω resistance of some materials in Table 8. SNL was also able to bring online an ABL system very late in the proficiency test, and found PETN to have sensitivity between what AFRL and IHD measured.

The relative ranking of the TIL values in Table 8 as well as the comparison with the group average values is difficult to accomplish because of the limited data available taken on the same ESD platform. However, as in Figure 5, LANL and IHD have complete data sets for all the materials on the same platform, which allows for a relative ranking between the two participants based on sensitivity:

LANL: $KClO_4$/Al > PETN = RDX 1 = RDX 3 = RDX 4 = GP = H_2O_2/Fl = H_2O_2/NM > RDX 2 = HMX > H_2O_2/Gl > H_2O_2/Cmn = AN/GP = $KClO_3$/Sg 100 > $KClO_3$/Sg AR = $KClO_3$/D = AN = UNi/Al = UNi/Al/S_8 > $KClO_4$/C = $NaClO_3$/Sg > $KClO_4$/D

IHD: $KClO_4$/Al > RDX 2 > RDX 3 > RDX 1 = RDX 4 > UNi/Al > $KClO_4$/D = HP/NM > $KClO_3$/D = $NaClO_3$/Sg = H_2O_2/Gl > GP > AN/GP > PETN = HMX > $KClO_3$/Sg 100 = $KClO_3$/Sg AR = $KClO_4$/C > AN = H_2O_2/Fl = H_2O_2/Cmn> UNi/Al/S_8

Other than KP/Al, there is little correlation between the two lists. Even using groupings based on sensitivity ranges, there are few if any matches.

Table 8. Average Threshold Initiation Level[1] Data for ABL ESD Testing[2-6]

Material[7]	LLNL, in J	LANL, in J	IHD, in J	AFRL, in J
RDX 1	NA	0.025	0.095	ND
RDX 2	NA	0.0375 ± 0.022	0.037	ND
RDX 3	0.038	0.025	0.076 ± 0.033	ND
RDX 4	0.038	0.025	0.095	ND
PETN[8]	0.033 ± 0.004	0.025	0.219 ± 0.093	0.043 ± 0.023
HMX	0.068 ± 0.006	0.0375 ± 0.022	0.219 ± 0.093	ND
$KClO_3$/Sg 100	NA	0.0625	0.272 ± 0.093	ND
$KClO_3$/Sg AR	NA	0.125	0.272 ± 0.093	0.092 ± 0.001
$KClO_3$/D	NA	0.125	0.142 ± 0.040	ND
$KClO_4$/Al	0.0088	0.0125	0.020 ± 0.005	0.025 ± 0.006
$KClO_4$/D	0.25	0.25	0.118 ± 0.040	0.273 ± 0.032
$KClO_4$/C	1.09 ± 0.30	0.208 ± 0.072	0.272 ± 0.093	0.13
$NaClO_3$/Sg	NA	0.208 ± 0.072	0.142 ± 0.040	0.177 ± 0.023
AN	NA	0.125	0.326	0.313 ± 0.058
GP	NA	0.025	0.1625	ND
AN/GP	NA	0.0625	0.165	ND
H_2O_2[9]/Cmn	NA	0.0625	0.326	ND
H_2O_2[9]/Fl	NA	0.025	0.326	ND
H_2O_2[9]/Gl	NA	0.038 ± 0.023	0.142 ± 0.040	ND
H_2O_2[10]/NM	NA	0.025	0.118 ± 0.040	ND
UNi/Al	0.323 ± 0.051	0.125	0.099 ± 0.064	ND
UNi/Al/S_8	0.25	0.125	0.791 ± 0.805	ND

1. Threshold Initiation Level (TIL) is the load (J) at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level; 2. Data from measurements using ABL system with 0-ohm resistance; 3. Data are the average of typically 3 measurements using 0/10 or 0/20 trials; 4. Values with ± indicate a range in results for the TIL trails, values with no ± indicate all results were the same; 5. NA = data not taken with 0-ohm resistance; 6. ND = not determined; 7. For abbreviations, see Table 1; 8. SNL measured 0.125 ± 0.043 J; 9. 70% H_2O_2 concentration; 10. 90% H_2O_2 concentration.

2.4.2 Comparison of ABL and Custom Built ESD

Table 9. ESD data from LLNL Custom and ABL Systems

Sample	Custom 510-Ω, TIL	ABL 0-Ω, TIL	Laboratory
RDX	0/10 at 1.0 J	0/10 at 0.038 J	LLNL
HMX	0/10 at 1.0 J	0/10 at 0.068 J	LLNL
PETN	0/10 at 1.0 J	0/10 at 0.033 J	LLNL
UNi/Al	0/10 at 1.0 J	0/10 at 0.038 J	LLNL
$KClO_4$/Al	0/10 at 0.25 J	0/10 at 0.088 J	LLNL

As mentioned above, LLNL started the proficiency test with a custom-built ESD system. This system was designed with a 510-Ω resistor in the circuit to mimic the human body. Several of the materials were tested with that system. Table 9 shows those results using the TIL method. During the proficiency test, LLNL obtained a new ABL ESD testing system and retested several of the materials. Table 9 shows these results and compares them to the ESD testing results by the custom-built system. The LLNL custom-built system indicates most of the materials to be insensitive to ESD. Only the $KClO_4$/Al mixture exhibits sensitivity to ESD. However, for the ABL systems, all laboratories reported measureable sensitivity of these same materials.

3 Discussion

Many of the figures show comparisons of the testing results on the same materials from the different participants. Clearly, there are some notable trends. It is important to note that these trends are not due to differences in the materials tested at each site. Each material was purchased by one participant from a single batch as specified by the manufacturer and distributed to all the other participants. The material was then pretreated as designated at the start of the proficiency test. Most of the materials studied are mixtures of two or more components, and the formulation and mixing procedures were also specified. In addition, some materials required specialized storage after pretreatment, which was also designated. These procedures are documented elsewhere [6,16]. Most pretreatment requirements were drying and sieving, and most mixing requirements were following component ratios and times from preparation to making measurements [4,16].

Although some effort was given to determine the nature of the differences in results among the participants, most of the reasons for these differences are not well defined. Much more research needs to be conducted on specific topics to link cause and effect that is beyond the scope of this paper.

3.1 Impact Results

3.1.1 Comparison of Individual Results

Figures 1 and 2 show that, compared to the average, LLNL drop hammer results show less sensitivity when testing is below 40 cm DH_{50} and LANL drop hammer results show less sensitivity when testing above 40 cm DH_{50}. This can be attributed to LLNL and LANL detection methods compared to the other participants. Both use microphones for positive detection, so detection sensitivity can potentially be more systematic than observation. However, the microphone type, placement and response factors are not standardized. The LLNL microphone system is less sensitive at low DH_{50} values, but is more affected by background noise at high DH_{50} values than the LANL microphone. AFRL values generally show each material has the highest sensitivity compared to corresponding values from other participants. This could be due AFRL using the largest striker weight and operator sensitivity. Also, IHD values generally track the LANL corresponding values but show higher sensitivity than LANL values. This is probably due to operator sensitivity compared to microphone sensitivity. IHD shared the design of the drop hammer with LANL, so they have almost the same equipment (different vintages, IHD—1945; LANL—1954).

Table 2 compares the relative rankings of impact sensitivity of the powdered materials. All four participants agree on the most sensitive materials. Except for AN, they agree on the insensitive materials also. AN is ranked with low sensitivity by all except LANL, where it is ranked with no sensitivity. AN has been shown to give a wide range of results because of the difficulty in determining a positive reaction [17]. The details of this will be reported elsewhere.

Table 3 compares the DH_{50} results for the mixtures containing H_2O_2. Different testing protocols were used for these materials than for the powders due to the physical nature of the samples. The principal issue is how to contain a material that can flow on the flat anvil surface. Using a bare anvil, as with LLNL and LANL, requires containment. Both used a ring of grease. This method, however, shows relatively insensitive results, probably because flat, finished surfaces (bare anvil and bare striker) provide few if no sites for reactions. Early work in impact testing has shown that abrasive action assists in initiating non-shock reactions [18], and the bare/smooth anvil does not have many of these sites. LLNL and IHD used 180-grit sandpaper for two of the mixtures. The results in the table reflect the increased number of abrasive sites of the sandpaper that facilitate reactivity as demonstrated by the increased sensitivity measured. IHD also used the Cavity Drop cell for some of the materials. The cell has flat surfaces touching the sample, but the sample is contained with an O-ring. The O-ring seals the sample into an airtight chamber. The chamber is filled 50% with the liquid sample and 50% with air. Adiabatic compression is the primary cause if there is initiation, although there may be small areas where the metal surface impacts the sample. The measured sensitivity is likely to be similar to the bare anvil. However, the design of the cell does not permit drop heights above 50 cm.

The only H_2O_2 mixture that shows significant sensitivity is the H_2O_2/NM. This is probably because the H_2O_2 component is 90% pure.

3.1.2 Sandpaper Effects

Table 4 shows that the relative values of the impact sensitivity of a solid material differ, depending upon what type of sandpaper is selected to hold the material on the anvil. This behavior varies much more for the HME materials than for the standard military materials.

To further highlight this difference, Table 10 shows results of impact testing of the $KClO_4$/Al mixture as performed by LANL, LLNL, IHD, and AFRL using different sandpapers. The values are averages of three or more determinations (listed in the supplementary material). All laboratories used 180-grit garnet sandpaper, and LLNL also used 120-grit Si/C sandpaper.

Table 10. Impact Testing Results for $KClO_4$/Al Mixture[1]

Laboratory	Sandpaper	DH_{50}, cm[2]
LLNL	120-grit Si/C	> 177
LLNL	180-grit garnet	16.9 ± 0.9
LANL	180-grit garnet	62.1 ± 6.8
IHD	180-grit garnet	41.3 ± 2.1
AFRL	180-grit garnet	41.4 ± 4.3

1. Each value average from three or more values listed in Supplementary material; 2. Modified Bruceton method, in cm, load for 50% probability of reaction (DH_{50}).

The average DH_{50} values based on sandpaper are: 120-grit Si/C, insensitive (exceeds equipment response); 180-grit garnet, 40.6 ± 15.8 cm (14 determinations). The standard deviation is below the 0.16 log unit range where applicable. For impact testing, when using 180-grit sandpaper, all participants show the $KClO_4$/Al mixture to be relatively insensitive. LLNL found it more sensitive than the LLNL-determined sensitivity of RDX while the other participants found the $KClO_4$/Al mixture value less sensitive than their determined sensitivity of RDX. This is further complicated by the LLNL determined sensitivity of the $KClO_4$/Al mixture using 120-grit sandpaper. LLNL could not measure a positive event in the drop hammer testing range using this sandpaper (LLNL drop height limit is 177 cm).

The results in Tables 10 and 4 show a substantial difference in DH_{50} values for a specific material when different sandpapers are used. This could be due to several factors seen in the properties of the sandpapers. Full analysis is beyond the scope of the proficiency test and will be reported in the future. Briefly, the types of sandpaper differ in several properties. 1) The different grit size means different average particle sizes (in mm: 120-, 150-, 180-grit; 0.115, 0.092, 0.082)[19]. This would suggest more particles/mm with smaller grit size, and therefore more reaction sites. However, this appears not the case because a visual inspection of SEM images indicates the particle counts are the same on all of the sandpapers (note: this count was hand done on a limited number of SEM images). 2) The grit composition also could be a factor. The 120-grit particle is Si/C and the 150- and 180-grit particles are garnet (general formula $X_3Y_2(SiO_4)_3$, X = several 2+ cations, Y = usually Al). 3) The hardness of the grit could be a factor because the values are different (garnet, 6.5-7.5; Si/C, 9-10, on the Mohs hardness scale [20]). This could greatly affect the number of actual reaction sites because the garnet may shatter more readily upon contact with the striker. Early work on non-shock initiation suggested that the harder the particulate, the better the chance for a reaction, although thermal conductivity cannot be ruled out [18]. 4) Sandpaper thickness and composition of the sandpaper backing may also play a role. The 120-grit paper is a woven fabric (consistent with wet/dry papers) with a thickness of 0.406 mm (0.016 in). The garnet papers are thin plain papers (consistent with dry only applications) with a thickness of 0.229 mm (0.009 in). This could affect energy transfer between the striker and the sample. 5) The glue could also affect the performance. The 120-grit sandpaper has a proprietary water resistant resin and the garnet papers have hide glue.

These other features of sandpaper, along with grit particles, must be included in a discussion of the role of sandpaper in non-shock initiated reactions. This subject has been studied for a long time, but almost all the studies focus on grit, and not the other aspects of the sandpaper. Clearly grit is important. Early work suggested grit hardness [21] as being a deciding factor, but more detailed studies indicated grit melting point and thermal conductivity were also important [22]. Unfortunately, the original studies did not represent hardness particularly well. Later studies have shown that grit interactions with metal surfaces cause hot spots (particularly relevant in skid tests), so hardness of the grit is important (with caveats of grit melting point and thermal conductivity), as well as particle size [23-27]. Although these studies are very important in understanding the effect of grit or dirt contamination on the stability of energetic materials, and include some sandpaper data, the definitive study on sandpaper still needs to be done. HME contributes additional issues as it can be considered grit in some cases (mixtures with solids) that has definite particle size ranges.

3.1.3 Neyer D-Optimal and Bruceton Method Comparison

Figure 3 compares the impact sensitivity (DH_{50}) of each of the materials as determined by the Neyer D-Optimal and the modified Bruceton methods. Note that these determinations are on separate testing data collected and reduced as opposed to the same testing data reduced by two different methods. For almost all the materials, these two methods give similar DH_{50} values for a specific material. However, PETN was the exception where the results from the two methods were considerably different: DH_{50} average three determinations, in cm, Neyer 10.3 ± 0.4; Bruceton 8.1 ± 0.5. This may be an anomaly in the proficiency test. LANL has extensive experience with a different preparation of PETN (RPS 3518) where the two methods give almost identical results averaged over several years: 3 recent years (n = 40) Bruceton, 13.8 ± 1.7 cm; 1.5 recent years (n = 36) Neyer 12.4 ± 1.9 cm. The measurements on the proficiency test PETN need repeating to understand the behavior.

3.1.4 Pressed Samples Compared to Powder Samples

Table 5 shows a limited data set on materials examined for impact sensitivity on both powder and corresponding pressed sample forms. Clearly pressing tends to make the material less sensitive to impact, consistent with early work on non-shock initiated reactions [28]. The choice of examining the HMEs as powders only was based on the unlikelihood of the materials ever being pressed in practice.

3.2 BAM Friction Sensitivity

3.2.1 Comparison of Individual Results

Figure 4 shows, for the BAM friction testing results, LLNL generally always has the highest F_{50} values compared to the other laboratories indicating LLNL finds each material less sensitive than LANL and IHD (note AFRL does not have BAM equipment and SNL limited data is not included). This is likely due to the LLNL system being completely enclosed in a glove box with a HEPA filter compared to other systems (see experimental below for the configuration). These added environmental and safety controls make determining a positive reaction more difficult because of the noise insulating effect of the glove box and the added background noise of the HEPA filter system. LANL sometimes has values lower than IHD that possibly is caused by room acoustics, operator differences, and/or humidity.

Table 6 shows the relative order based on the F_{50} values of sensitivity. As noted, LLNL and LANL have similar rankings due to sensitivity, and, if the H_2O_2-based materials are removed from the list, IHD also agrees, in general. Both LLNL and LANL measured the H_2O_2 mixtures to be completely insensitive to the friction stimulation. The reasons for this have not been elucidated, but are suspected to be due to the ceramic sample holder and the stylus used for stimulation not being standardized for the proficiency test or also due to humidity effects. The stylus used by LANL, for example, was purchased from a different source than the stylus used by LLNL. As far as humidity effects, the ceramic sample holder is dried before use, but the differences among ambient relative humidity at the three participants (IHD, approximately 50%; LANL, < 10%; LLNL, 10-25%) could effect how much the porous ceramic absorbs moisture before analyzing the sample.

The friction sensitivities as measured by the TIL method, shown in Table 7, for the most part follow the relative order or sensitivities as measured by the F_{50} method. IHD found more of the H_2O_2 mixtures sensitive, while LLNL and LANL did not. This result, combined with the behavior seen in the F_{50} data suggests that IHD system responds to viscosity differently than the LLNL and LANL systems for an undetermined reason.

3.3 ESD Sensitivity

3.3.1 ABL ESD Sensitivity

Figure 5 shows for the ESD testing, IHD reports materials to be less sensitive compared to corresponding values determined by LANL. This could be due to IHD having the oldest ABL ESD equipment of the participants, using different needles, operating in higher humidity, and operation with a detection procedure with the room lights on vs. off. The IHD instrument was one of the first brought into production and has not had the same calibration as the newer equipment. The absolute values of these energy levels are likely different than later-built equipment. In addition, the age of the instrument is reflected in the lack of flexibility in setting energy levels, something very important in TIL measurements. The supplementary material also lists the relative humidity during the measurements for all the participants. IHD levels are always close to 50% RH, while LANL levels are < 10% except for two months in the late summer. Humidity has been known to affect static discharge [29]. Note, in these determinations, AFRL measured a limited subset of materials, and most of LLNL data is from the custom built system that was not included in the figure.

3.3.2 Comparison of ABL and Custom ESD sensitivity

Table 9 compares selected ESD sensitivity determined by the TIL method from data taken by LLNL on the ABL and the custom-built systems. The 510-Ω resistor in the circuit of the custom-built system has a very large affect on the indication of sensitivity. Only $KClO_4$/Al of the entire suite of materials measured by LLNL had any sensitivity with this system. The 510-Ω resistor is a more practical assessment of electrostatic transfer through the body, which was the design criteria of the system. NIOSH has determined the human body response varies depending upon the energetic bias placed on it, but uses 1000 Ω as a guideline for the human body resistance [30]. While resistance can be added to new versions of the ABL system, older systems, such as those at LANL and IHD do not have this capability. As a result, 0-Ω resistance was used as the baseline setting.

3.4 Parameter Variations

The proficiency test sample suite presents several permutations in materials and/or test conditions. These variations were chosen to see if certain material properties (such as a series of oxidizers) or if test conditions (such as sandpaper type) can make a difference in SSST results. These variations are:

1. RDX Standard tested 4 times or more
2. 10+ materials where DH_{50} values measured at two or more types of sandpaper (120-, 150-, and 180-grit)
3. 20+ materials analyzed by two DH_{50} analysis methods (Neyer and Bruceton)
4. 20+ materials analyzed by two friction analysis methods (threshold (TIL) and Bruceton (F_{50}))
5. 10+ materials analyzed on two ESD apparatuses (ABL and LLNL Custom)
6. H_2O_2 mixtures with two different concentrations of H_2O_2 (90 and 70 %)
7. H_2O_2 mixtures with four different organic fuels
8. 18+ materials measured in DSC with two sample holders (vented and sealed)
9. 20+ materials analyzed on two different friction apparatuses (ABL and BAM)
10. 2 mixtures with particle size differences in $KClO_3$
11. 2 fuels (sugar and dodecane) mixed with three different oxidizers ($KClO_3$, $KClO_4$, $NaClO_3$)
12. 1 oxidizer ($KClO_4$) mixed with three different fuel types (Al, C, and dodecane)
13. 2 component mixture combinations (AN, Gunpowder, AN/Gunpowder; UNi/Al and UNi/Al/S_8).

The data for variation **1** are presented here in summary form. However, the data have been reduced using statistical methods and the results will be published separately. Variations **2-7** are discussed above. Variations **8** and **9** will be published separately. Variations **10-13** are discussed below.

Variation **10**—$KClO_3$/Sg 100 and $KClO_3$/Sg AR mixtures differ based on particle size of the $KClO_3$ [31]. The $KClO_3$ in $KClO_3$/Sg 100 mixture was separated using a 100-mesh sieve; in $KClO_3$/Sg AR was used as received (all went through a 40 mesh sieve). Figure 6 shows the pore size distribution and photographs of the two preparations of $KClO_3$ used in the $KClO_3$/Sg mixtures. Except for large particles in the AR photos, there are little differences in the size distributions. Likewise, Table 11 shows there is little difference in the SSST results for the two mixtures when comparing intra-laboratory results.

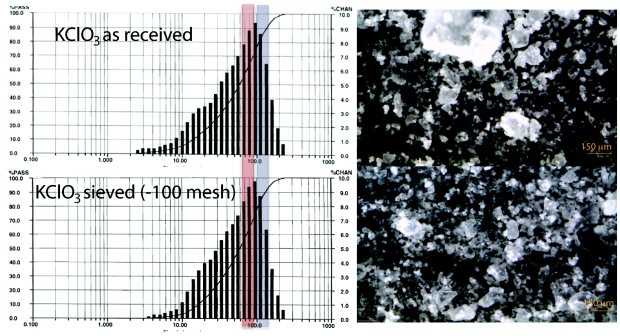

Figure 6. Size distribution by laser light scattering (left side) and photograph (right side) of $KClO_3$ as received and screened through a 100-mesh sieve.

Table 11. Average SSST Testing Results for $KClO_3$/Sg 100 and $KClO_3$/Sg AR

	LLNL	LANL	IHD
Impact	*DH_{50}, cm*	*DH_{50}, cm*	*DH_{50}, cm*
$KClO_3$/Sg AR	15.6	10.3	10.3
$KClO_3$/Sg 100	14.9	10.6	14.3
Friction	*TIL, kg; F_{50}, kg*	*TIL, kg; F_{50}, kg*	*TIL, kg; F_{50}, kg*
$KClO_3$/Sg AR	9.5; 11.8	2.4; 4.9	3.2; 3.6
$KClO_3$/Sg 100	6.9; 9.9	4.8; 5.8	2.3; 4.4
ESD	*TIL, Joules*	*TIL, Joules*	*TIL, Joules*
$KClO_3$/Sg AR	0/10 at 1.0	0/20 at 0.125	0/20 at 0.272
$KClO_3$/Sg 100	0/10 at 1.0	0/20 at 0.0625	0/20 at 0.272

Impact sandpaper, LLNL 120-grit, LANL and IHD, 180-grit; TIL for BAM friction, 0/10 for LLNL; 0/20 for LANL and IHD; ESD LLNL Custom built; LANL and IHD ABL

As shown above, sandpaper grit particulate size possibly has some effect on determining the sensitivity of a material, certainly in drop hammer studies. It does not appear that this extends to the particle size of the materials themselves. However, Figure 6 shows that both the particle size and distribution of the two materials are about same, except for very large particles. Because the sampling is so small in SSST testing, a potential reason for the similar DH_{50} sensitivities of the two mixtures could be that the sampling selected about the same size materials and left out the large size material, and therefore the testing gave similar results. The probable hidden parameter is there is no particle size dependency for these two mixtures.

Variation **11**—Mixtures of sugar with $KClO_3$ and $NaClO_3$ [32] and dodecane with $KClO_3$ and $KClO_4$ [33] were examined to see if the oxidizer had much affect on the sensitivity of the mixture. Table 12 compares the SSST results between $KClO_4$/D and $KClO_3$/D for LLNL, LANL, and IHD. Clearly the oxidizer does make a difference in the results for impact and BAM friction sensitivity. Not shown is the comparison between $NaClO_3$ and $KClO_3$ mixtures with sugar (raw data can be found in the supplementary material). Similarly, when comparing intra-laboratory results, the $NaClO_3$/Sg mixture was less sensitive to impact, yet similar in sensitivity to BAM friction, and ESD compared to the corresponding results for the $KClO_3$/Sg AR mixture.

Table 12. Average SSST Testing Results for $KClO_4$/D and $KClO_3$/D

	LLNL	**LANL**	**IHD**
Impact	*DH_{50}, cm*	*DH_{50}, cm*	*DH_{50}, cm*
$KClO_4$/D	30.5	28.8	19.3
$KClO_3$/D	9.3	8.1	10.3
Friction	*TIL, kg; F_{50}, kg*	*TIL, kg; F_{50}, kg*	*TIL, kg; F_{50}, kg*
$KClO_4$/D	>36; >36	>36; >36	33; >36
$KClO_3$/D	12.3; 25.5	7.2; 19.1	16.5; 26.8
ESD	*TIL, Joules*	*TIL, Joules*	*TIL, Joules*
$KClO_4$/D	0/10 at 1.0	0/20 at 0.250	0/20 at 0.118
$KClO_3$/D	0/10 at 1.0	0/20 at 0.125	0/20 at 0.140

All DH_{50}, 180-grit garnet sandpaper; BAM friction, TIL 0/10 for LLNL, 0/20 for LANL and IHD; ESD LLNL Custom Built, LANL and IHD, ABL

Table 13. Average SSST Testing Results for $KClO_4$/Fuel Mixtures

	LLNL	**LANL**	**IHD**
Impact	*DH_{50}, cm*	*DH_{50}, cm*	*DH_{50}, cm*
$KClO_4$/C	> 177	> 320	> 320
$KClO_4$/Al	17	62	41
$KClO_4$/D	30.5	28.8	19.3
Friction	*TIL, kg; F_{50}, kg*	*TIL, kg; F_{50}, kg*	*TIL, kg; F_{50}, kg*
$KClO_4$/C	> 36; >36	> 36; >36	> 36; ND
$KClO_4$/Al	12.3; 25.5	7.2; 19.1	16.5; 26.8
$KClO_4$/D	>36; >36	>36; >36	33; >36
ESD	*TIL, Joules*	*TIL, Joules*	*TIL, Joules*
$KClO_4$/C	0/10 at 1.0	0/20 at 0.250	0/20 at 0.118
$KClO_4$/Al	0/10 at 1.0	0/20 at 0.125	0/20 at 0.140
$KClO_4$/D	0/10 at 1.0	0/20 at 0.250	0/20 at 0.118

Impact sandpaper, LLNL 120-grit, LANL and IHD, 180-grit; BAM friction TIL 0/10 for LLNL, 0/20 for LANL and IHD; ESD LLNL Custom Built, LANL and IHD, ABL

Table 14. Average SSST Testing Results for AN, GP, and AN/GP

	LLNL	**LANL**	**IHD**
Impact	*DH_{50}, cm*	*DH_{50}, cm*	*DH_{50}, cm*
AN/GP	46.8	29.0	21.3
GP	54.2	20.7	12.3
AN	82	> 320	201
Friction	*TIL, kg; F_{50}, kg*	*TIL, kg; F_{50}, kg*	*TIL, kg; F_{50}, kg*
AN/GP	27.0; 32.7	13.0; 19.0	12.2; 12.7
GP	16.4; 20.7	5.6; 9.3	13.8; NA
AN	> 36; > 36	> 36.7; > 36.7	> 36.7; > 36.7
ESD	*TIL, Joules*	*TIL, Joules*	*TIL, Joules*
AN/GP	0/10 at 1.0	0/20 at 0.0625	0/20 at 0.165
GP	0/10 at 1.0	0/20 at 0.0250	0/20 at 0.1625
AN	0/10 at 1.0	0/20 at 0.125	0/20 at 0.326

All DH_{50}, 180-grit garnet sandpaper; BAM friction TIL 0/10 for LLNL, 0/20 for LANL and IHD; ESD LLNL Custom Built, LANL and IHD, ABL

Variation **12**—$KClO_3$ was mixed with three different fuel sources to see the effects certain types of fuel have on sensitivity [34]. Dodecane and C (as activated charcoal) are organic based materials; Al was selected as an inorganic component (added to energetic materials for increased afterburn). Table 13 shows the comparisons of the average results. The big differences are in the impact and the friction sensitivity, as the C mixture shows insensitivity. For ESD, however, the fuel source is sensitive to spark and possibly drives the results.

Variation **13**—AN and GP were tested separately and then combined and tested [35]. Table 14 shows the average results. Interestingly, the AN/GP sensitivities are not linear combinations of the two component sensitivities.

Synergetic affects between the GP and AN are likely the cause of this. However, AN is an extremely difficult material to subject to SSST testing [36], especially for the drop hammer experiment. It has low sensitivity and is difficult to discern a positive reaction (a more detailed account of this will be presented elsewhere). Another issue is sampling. DSC studies have shown it is difficult to obtain a representative sample because of the difference in particle size distribution. Figure 7 shows the particle size distribution for the two component materials. The GP is much more narrow in the distribution and if small samples are obtained, the sample can easily not match the bulk compositionally. Also studied were the UNi/Al and UNi/Al/S_8 mixtures. These mixtures exhibited almost no sensitivity, so a comparison of the effects of adding sulfur cannot be assessed.

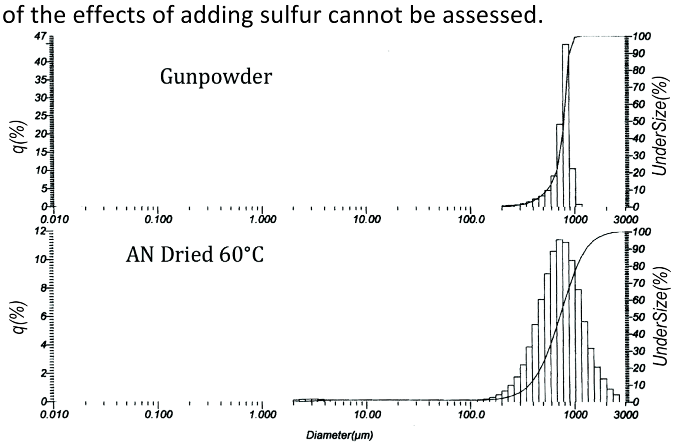

Figure 7. Particle Size distribution of GP and AN

4 Experimental Section

4.1 Materials

4.1.1 Sources

Table 1 lists the materials used in this study. The mixtures were prepared according to IDCA procedures [6]. Each material came from a common batch that was distributed at the beginning of the program to each participant. Except for H_2O_2, which was distributed by the purchasing laboratory to the participants, because of safe handling requirements by the manufacturer (FMC). The batch was portioned by the manufacturer and then distributed to the participants (FMC required special handling procedures and training). The H_2O_2 was received as 90% and 70%; concentrations checked by refractive index and used directly [40]. The RDX Type II Class 5 standard was lot # HOL89D675-081; the PETN Class 4 was lot # W723220, both manufactured by Holston Army Ammunition plant and both donated by IHD. In the case of mixtures, the components were distributed then mixed at the testing site. The following chemicals were purchased: $KClO_4$ and $KClO_3$ from Columbus Chemicals Industry; $NaClO_3$, AN, nitromethane, and glycerine from Fisher Scientific; Bullseye® smokeless powder from Alliant; UNi from TCI America; Al from Valimet; charcoal from Aldrich; dodecane from Alfa Aesar; S from Sigma-Aldrich; icing sugar from C & H; flour from Piggly

Wiggly (Laxmi brand); cumin from Safeway (generic), C (as activated DARCO brand charcoal) from Sigma-Aldrich, and HMX from ATK.

4.1.2 Pretreatment, Storage and Mixing

Details of sample pretreatment, storage and mixing are described in detail elsewhere [6,16]. Briefly, RDX, $KClO_3$, $KClO_4$, $NaClO_3$, sugar, cumin, flour, AN, and C were dried at 60°C for 16 hours then cooled and stored in a dessicator until use. $KClO_3$, sugar, cumin and flour materials were separated by sieves before pretreatment. RDX, AN and C were dried at each laboratory, while cumin and flour were separated by sieve (100 mesh), dried, then distributed.

All materials were mixed at 10 g or less. The solid-solid mixtures were blended by hand (teflon spatula) for 5 to 10 min and then mixed using a jar mill or V-blender for and additional 5-10 min. Solid-liquid mixtures (except H_2O_2 mixtures) were blended for 5 to 10 min until homogeneous by hand or with a magnetic stirring system. The liquid-liquid systems (H_2O_2 mixtures) were mixed using a magnetic stirring system for 10-15 min while the temperature was monitored with thermocouple. All mixtures were used after a 1-h rest period. ***Safety note***: H_2O_2 mixtures have been documented to self-heat with time at larger batch sizes. It is recommeded to use the mixtures within a few hours of mixing and then destroy the residuals. If longer term experiments are necessary, monitor the temperature profile for the on-set of run away reactions [37].

4.2 Testing Methods

For the Proficiency test, the SSST testing methods were alligned as much as possible without signifcantly modifying the standard test procedures incorporated at each laboratory. There were slight differences in procedures, but when possible, these procedures were standardized (such as distributing the same sandpaper for drop hammer testing) if the change had little or no deviation from the adopted procedure for each laboratory.

4.2.1 Impact Testing

Impact testing was performed with custom built equipment at LLNL, LANL, and IHD as Type 12, and with commercial equipment at AFRL and SNL as modified Bureau of Mines (MBOM) to Type 12 testing. Figure 8 shows exmples of both systems (LLNL and AFRL).

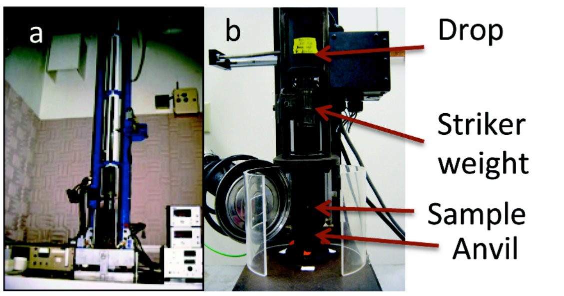

Figure 8. Examples of drop hammer equipment: a) Type 12 at LLNL (circa 1956); b) MBOM with Type 12 tooling at AFRL (circa 2009).

For the drop hammer testing of solids, the sample size was 35 ± 2 mg; 180-grit garnet dry, 150-grit garnet dry and 120-grit Si/C wet/dry sandpaper were used to hold sample in place; drop weight was 2.5 kg; striker weight was variable (LLNL, IHD, AFRL and SNL 2.5 kg, LANL 0.8 kg); positive reaction was a pop, flash or smoke; detection was by sound meter (LLNL, LANL) and/or observation (LLNL, LANL, IHD, AFRL, SNL). For drop hammer testing of liquids, these apply: sample held by grease ring (LANL, AFRL, SNL), grease ring or sandpaper (LLNL), grease ring, cavity drop or sandpaper (IHD); drop weight 1.0 kg (LLNL only).

4.2.2 BAM Friction Testing

Friction testing was performed with BAM friction test equipment of various vintages, but all of essentially the same design.

Figure 9 [41] shows examples of the different configurations of the BAM friction equipment. For the testing, the sample sizes ranged from 5 to 40 mg (approximated but not weighed); samples were held on a ceramic plate that varied in size and composition; pin on which the load is applied also varied in composition; environmental control with a suction duct (IHD, LANL) or with a closed glove box and HEPA filter (LLNL); detection for a positve event is by observation for all participants of a pop, smoke, or jetting.

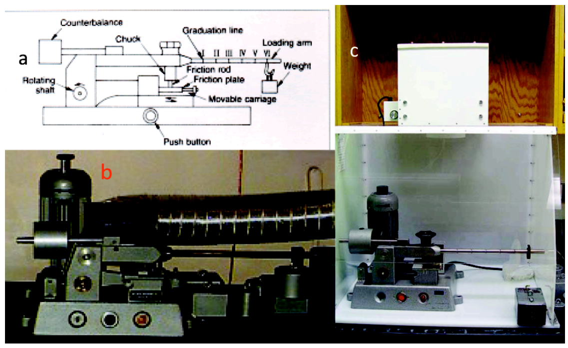

Figure 9. Examples of BAM friction equipment: a) diagram, b) IHD venting configuration, c) LLNL venting configuration.

4.2.3 ESD Testing

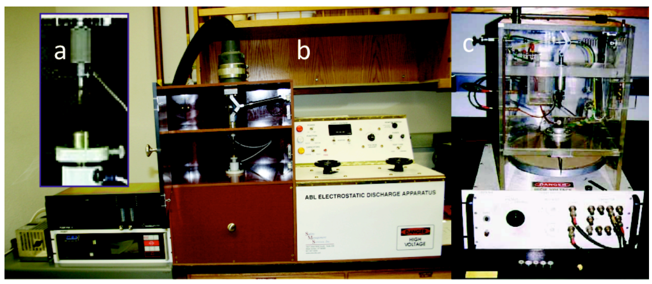

Figure 10. Examples of the ESD equipment used in the proficiency test: a) close-up of ABL electrodes, b) ABL equipment, c) custom-built equipment by LLNL.

For the proficiency test, all participants employed ABL ESD testing equipment of different vintages. In addition, LLNL utilized a in-house custom built system with a 510-Ω resistor in series in the circuit to mimic the human body. The test is simple, the sample is placed between two electrodes and a charge that is stored in a capacitor is discharged through the sample. Figure 10 shows examples of the ESD equipment utilized by the participants. Generally, the sample size was 5 to 40 mg (approximated but not weighed); the sample was held in place by tape (LANL-scotch, LLNL-Mylar) or none (IHD, AFRL); detection by observation for all participants; positive detection by a pop, puff, smoke, noise. Energy is set at discrete levels as dictated by the version of the ABL ESD and the custom-built system.

4.3 Data Reduction Methods

For impact testing, two methods were used—modified Bruceton [7,38] and Neyer D-Optimal [8]. For friction testing, three methods are used—modified Bruceton, Neyer D-Optimal (rarely), and TIL (threshold) [9]. The D-Optimal method was applied to friction data in two cases at the high end of insensitivity because test conditions no longer met the modified Bruceton assumptions test criteria. For ESD testing, one method was used—TIL. These methods have been reviewed previously, are well established and will only be summarized briefly here.

4.3.1 Threshold Initiation Level (TIL)

This method [9] tests at discrete energy levels and determines the level that gives no reaction in a set amount of trials as well as levels that trigger reactions. For BAM friction, the TIL is the load (kg) at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level. In this BAM friction test, the load levels are controlled by the position and size of the weight positioned on the load arm. For ESD, the TIL is the load (joules) at which zero reactions out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level. In ESD, the load level is set by the energy (which is converted to joules at a fixed voltage) charged to the circuit to generate the spark that passes through the sample.

4.3.2 Probability of Reaction

Both the modified Bruceton and the Neyer D-Optimal are employed to determine 50% probability of reaction. For the proficiency test, all laboratories used the modified Bruceton method, and LANL also used the Neyer D-Optimal method. The general approach of the two methods is the same: choose an algorithm for picking test levels and number of tests, carry out testing, following the algorithm, noting the result at each level, and then analyze the results for mean and standard deviation. An optimized algorithm meets laboratory and testing needs: faster determination with fewer tests, better determination of mean (higher confidence), and better determination of standard deviation. However, there are drawbacks in this approach. Attempts to optimize all of these simultaneously usually leads to trading confidence for more rapid testing (fewer tests).

The modified Bruceton method was developed at ERL, Bruceton PA, in early 1940s, and was optimized by Tukey [39], Dixon, and Mood [7]. It is optimal for determining 50% reaction level, but is not optimal for determining standard deviation without extra testing. The Neyer D-Optimal method was developed at EG&G Mound, Miamisburg, OH in the 1980s by Barry T. Neyer [8]. It does not rely on very strict assumptions, and can run tests optimized for mean or some other probability level—at any positive or negative test level. The result is equal confidence in mean and standard deviation.

4.3.3 Averaging Methods

Almost all determinations (experimental data sets for each data reduction method) were performed in triplicate (or more). The Bruceton and Neyer methods produce a value that represents 50% probability of reaction (midway on a reactivity curve) and a standard deviation. The individual determinations were averaged and these values were used for the graphs and the comparisons in this report. The TIL method determines the onset of the sensitivity (approximately 2 or 3% on the upswing of the reactivity curve). This uses a discrete insult level approach, so only specific levels are recorded in the raw data. These specific levels correlate to limited settings on the equipment. As a result, because each participant had different vintages of the same equipment, each participant reported different, but discrete levels in the raw data. Although somewhat of an inexact method, the three (or more) discrete results for a specific material were averaged for the comparisons. This can result in a value that the participant can not actually measure. For example, ESD data for HMX by IHD is 0.165, 0.326, 0.165 J, which are discreet settings of the ABL equipment used by IHD for the measurements. In this report, the three values are averaged to 0.219 J, which is not a setting on the IHD equipment. However, this estimates the average insult level determined by IHD in the ESD comparison figure.

5 Conclusions

Many of the HME materials tested in this study are sensitive to the differences in the test methods and equipment employed by each laboratory. This leads to differing evaluations of sensitivity, some of which are significant from a safety standpoint. Some of these differences can be eliminated by standardization but others are inherent in the configurations and environments that each laboratory has established to safely test energetic materials. Elimination of the differences will require further research to accomplish, however. The work has shown that it is important to be able to test materials under a variety of conditions because of the multiple types of insults possible to these materials. Exploring a range of variables provides the best chance of probing the particular set of test parameters that highlight the extent of sensitivity of the material. Sandpaper properties, striker mass, and the method of detecting the generated sound or reaction are all examples of important variables. Because these variables may not be easily explored at a single facility, it is very beneficial, if possible, to have materials tested at multiple laboratories and reported in the literature. The established standards that each laboratory uses are still appropriate for testing HME materials because they generally show that an instrument and method are working as expected, as long as material is well characterized and the standards are well studied.

Symbols and Abbreviations

ABL—Allegany Ballistics Laboratory
AFB—Air Force Base
AFRL—Air Force Research Laboratory, Tyndall AFB, FL
AN—Ammonium nitrate
AR—As received
BAM—German Bundesanstalt für Materialprüfung
DH_{50}—The height the weight is dropped in the drop hammer experiment that cause the sample to react 50% of the time, calculated by the Bruceton or Neyer methods
DHS—Department of Homeland Security
DSC—Differential Scanning Calorimetry
ESD—Electrostatic Discharge
F_{50}—The weight used in friction test that cause the sample to react 50% of the time, calculated by the Bruceton or Neyer methods
HEPA—High efficiency particulate air filter
HMEs—Home made explosives (improvised explosives)
HMX—Cyclotetramethylene-tetranitramine
IDCA—Integrated Data Collection and Analysis Program
IHD—Naval Surface Warfare Center, Indian Head Division
LANL—Los Alamos National Laboratory
LLNL—Lawrence Livermore National Laboratory
MBOM—Modified Bureau of Mines drop hammer
NIOSH—National Institute of Occupational Safety and Health
PETN—Pentaerythritol tetranitrate
RDX—(1,3,5-Trinitroperhydro-1,3,5-triazine)
SEM—Scanning Electron Microscope
SNL—Sandia National Laboratories
SSST—Small Scale Safety and Thermal Testing
TIL—Threshold Initiation Level
UNi—Urea nitrate

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[38] The Bruceton method also assumes that testing begins in the vicinity of the mean. Often this is not true and the initial testing to home in on the mean can skew the statistics. In practice, a "Modified" Bruceton method is used in which initial tests are used to bracket the mean before starting to count Go's and No-Go's. This is used by LANL in this work.

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